CHARGE NUMBER: 2500

PROGRAM TITLE: SYNTHESIS OF TOBACCO ADDITIVES

PERIOD COVERED: May 1 - May 31, 1973

PROJECT LEADER: S. C. Clough

DATE OF REPORT: June 4, 1973

I. FLAVOR RELEASE PROGRAM

The custom synthesis and purification of ethyl α,α-dimethyl-β-hydroxy-β-(4-methoxyphenyl)propionate (CR-1214) have been completed, and the material has been delivered to Flavor Development.[1] Thermal degradation of CR-1214 has been shown to occur at 270-275°C (DTA, DSC) under static conditions.[2] The thermal degradation products, the feasibility of scaleup procedures, and the definition of the purity specifications for this cherry flavor release compound are currently being investigated.[3]

II. NICOTINOID CHEMISTRY

The synthesis of 2',3',3',4',4',5'-hexadeutero-5'-carbomethoxynicotine was completed for a study which shows the involvement of a pyridyl proton in the mass spectral fragmentation pattern. This completed current efforts towards a paper to be submitted to "Organic Mass Spectrometry."[4]

Two new compounds, 5'-carbomethoxynornicotine (CR-1298) and 5'-carboxamidonornicotine (CR-1299) were prepared for use as potential intermediates in the synthesis of 5'-cyanonicotine.[4]

N-cyclopropylnicotinaldehydeimine was prepared as a potential precursor of 2(3-pyridyl)-3,4-dihydro-2H-pyrrole.[5] If the latter is stable, it would serve as an intermediate to 5'-substituted nicotines; if the double bond spontaneously shifts into conjunction, this synthesis represents a one-step route to myosmine.

Improvements were made in the procedure for the methylation of 2-(3-pyridyl)azetidine and a larger quantity of the methyl analog was prepared.[6] The d,l-pyridylazetidines (methyl and nor), d,l-nicotine, and d,l-nornicotine are to be shipped to Woodard Research Corporation for testing purposes.

III. REFERENCES

1. Memo from E. G. S. Rundberg, Jr. to T. E. Majewski, May 14, 1973.

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2. W. Ryan, Notebook 6154.
3. E. Rundberg, Notebooks 6217 and 6327.
4. J. DeBardeleben, Notebook 6216.
5. W. Edwards, III, Notebook 6200.
6. H. Secor, Notebook 6195.

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